USE OF ICP-AES OR ICP-MS TO DETERMINE AQUEOUS CONSTITUENT CONCENTRATIONS

PROCEDURE ID: YMP-LBNL-TIP/AFT 11.0 | REV.0, MOD. 0 | EFFECTIVE: 06/30/2000

1. PURPOSE

This Technical Implementing Procedure (TIP) provides instructions to determine, using Inductively Coupled Plasma–Atomic Emission Spectrometry (ICP-AES) or Inductively Coupled Plasma–Mass Spectrometry (ICP-MS), the concentration of chemical constituents in liquid samples at Lawrence Berkeley National Laboratory (LBNL) for supporting the Yucca Mountain Site Characterization Project (YMP).

2. SCOPE

This procedure applies to all LBNL personnel (or contractor personnel following LBNL procedures) involved in the calibration and use of instruments (ICP-AES or ICP-MS) for detection and quantification of constituents of interest in solution, except when other procedures (e.g., YMP-LBNL-TIP/TT 13.0) are in effect. Prior to conducting work described in Section 3 of this procedure, personnel performing measurements require training to this procedure.

All technical activities, data collected using this procedure and any equipment calibrations shall be in accordance with this TIP and in full compliance with YMP Administrative Procedure (YAP)-12.3Q, *Control of Measuring and Test Equipment and Calibration Standards*. All documentation resulting from actions taken under this TIP shall be recorded in Scientific Notebooks and/or Equipment Logbooks (which are controlled as supplemental records) as described in the Office of Civilian Radioactive Waste Management (OCRWM) Administrative Procedure (AP)-SIII.1Q, *Scientific Notebooks*. While this procedure incorporates specific requirements unique to the laboratory studies, it is consistent with the requirements described in YMP-LBNL-QIP-SII.0, *Documenting Sample Control*.

If this procedure cannot be implemented as written, YMP-LBNL personnel shall notify the responsible Principal Investigator (PI) or designee. If it is determined that a portion of the work cannot be accomplished as described in this TIP, or would produce undesirable results, that portion of the work shall be stopped and not resumed until this procedure is modified per YMP-LBNL-QIP-5.2, *Preparing Development Plans & Quality /Technical Implementing Procedures*.

If the responsible PI or designee determines that a modification or a revision to the TIP would cause an unreasonable delay in proceeding with the task, then an expedited change to the procedure, including documentation of deviation from the approved procedure, can be made according to YMP-LBNL-QIP-5.2. Such changes are subject to

review, usually after the task has proceeded, and thus work performed under TIPs with expedited changes is done at risk of future invalidation.

Employees may use a controlled electronic or hard copy of this procedure; however, employees are responsible for assuring that the correct revision of this procedure is used. When this procedure becomes obsolete or superseded, it must be destroyed or marked "superseded" to ensure that this document is not used to perform work.

3. PROCEDURE

3.1 Analytical Techniques and Samples

3.1.1 Analytical Techniques

ICP-AES determines trace elements, including metals, in solution. Environmental Protection Agency (EPA) Solid-Waste Test Methods (SW-846) Method 6010B "Inductively Coupled Plasma-Atomic Emission Spectrometry" (Revision 2, December 1996, See Attachment 1) shall be used to employ ICP-AES for the chemical analysis. The Method 6010B is applicable to all of the elements (including sodium, silica, calcium, potassium, lithium, aluminum, magnesium, iron, and manganese) listed in Table 1 of the Attachment 1. ICP-MS is applicable to the determination of sub-ug/L concentration of elements listed in Table 1 of EPA Method 6020 "Inductively Coupled Plasma-Mass Spectrometry" (Revision 0, September 1994, See Attachment 2). For example, if adequate sensitivity is not obtained for aluminum using the Method 6010B, Method 6020 could be used to determine aluminum concentration at the discretion of the responsible PI or designee.

3.1.2 Samples

Liquid samples that require chemical analysis may be from various sources, including (but not limited to) laboratory and field tracer work with well-defined objectives, and water observed in the field that chemical analysis will potentially help to elucidate flow and transport processes.

A. Sample Name/Bottle Labeling

Samples shall be collected in the appropriate containers (e.g., high-density polyethylene bottles with tight sealing caps) deemed suitable for collection and storage of samples. Under certain conditions, collection pads could also be used to collect samples (e.g., seepage water). Care shall be taken (e.g., wear gloves) to prevent cross-contamination during sample collection. Each sample shall be given a unique identifier to reflect the sample source or an appropriate

abbreviation thereof. Sample names shall be marked with an indelible marker either directly on the bottle or on an adhesive sticker affixed to the bottle along with the name of the originator and the date. The sample name, time and location of sample collection, collection method, and the unique identifier assigned by the YMP Sample Management Facility (SMF) (applicable if the sample is collected in the field site, in accordance with YAP-SII.1Q *Submittal, Review, and Approval of Requests for Yucca Mountain Site Characterization Project Geologic Specimens*) shall be entered into the scientific notebook.

Safety considerations associated with handling of chemicals will depend on the chemical nature of the constituents in the solutions. Material Safety Data Sheets (MSDSs) shall be consulted to determine whether special protective clothing and/or eye protection are required. A hazard label shall be placed on any sample bottle that contains hazardous chemicals.

B. Sample Handling/Preservation

Following sample collection, samples shall be refrigerated whenever possible for later delivery to the Environmental Measurements Laboratory (EML) at LBNL (or other analytical laboratories) for analysis. For samples collected at field sites, refrigeration after sample collection and during sample transfer to LBNL may not be feasible. At the discretion of the responsible PI or designee, alternative steps (e.g., putting the ice packs together with the samples during sample storage and transfer) shall be taken to mitigate the potential sample degradation. The method of preservation shall be recorded in the scientific notebook.

The samples shall be analyzed within six months after collection (U.S. Environmental Protection Agency, 1996, Chapter 3). If samples cannot be analyzed within this timeframe, they shall be analyzed at the first available opportunity, and a notation shall be placed in the scientific notebook identifying the duration (obtained from sample collection date recorded on the collection bottle, and the analysis date) samples have exceeded the analysis timeframe. A notation shall also be placed on the analysis results allowing special consideration to be given to data generated, and an analysis of the data applicability. This analysis shall be documented in the scientific notebook.

C. Sample Transfer

Samples shall be transferred to the EML along with a chain-of-custody form (Attachment 3) containing the sample identification, time of collection, and analyses required. A copy of the chain-of-custody form

shall be placed in the scientific notebook, and a copy shall be left with the samples. Signatures of the person delivering the samples and the person receiving the samples, and the time and date of transfer shall be placed on the chain-of-custody form. The EML may assign another tracking number to the samples if necessary, and this identification shall be placed on the chain-of-custody form next to the originally given name to provide traceability.

3.2 Calibration and Sample Analysis Procedure

3.2.1 Identification of Standards to be Used

Calibration standards for ICP-AES and ICP-MS shall be traceable to nationally recognized standards [e.g., National Institute of Standards and Technology (NIST)]. Verification of NIST traceability shall be demonstrated by a certificate of analysis (to be filed in the scientific notebook) provided by the manufacturer or vendor showing the comparison of the standard purchased to the NIST standard. The calibration standards shall be procured from YMP-approved contractors on the Qualified Suppliers List (QSL), or an alternative approach such as Activity Evaluation shall be pursued according to AP-2.16Q, *Activity Evaluation*. The Measuring and Test Equipment (M&TE) Justification form shall be documented for each standard used to calibrate M&TE in accordance with YAP-12.3Q. This form shall be filed in the scientific notebook.

Additionally, a second NIST-traceable standard of similar quality obtained from a different manufacturer will be used to verify calibration standards as required by Section 5 of Attachments 1 and 2. Thus, two independent standards will be used, one in calibrating the equipment, and the other in verifying the calibration. The verification standards are not necessarily purchased from YMP-approved contractors on the QSL.

3.2.2 Preparation of Standards

Procure the concentrated stock solutions for both calibration and verification standards (see Section 3.2.1) with their concentrations stated (e.g., 1,000 mg/L). Prepare, by serial dilution, a set of calibration standard solutions that span the expected range of concentrations in the samples or the instrument response. For each dilution step, use the pipette and/or pipettor to take the appropriate volume (e.g., 10 ml) of concentrated stock solution into a volumetric flask (e.g., 100 ml) and fill the water to the calibration line of the flask. Mix the solution thoroughly. For example, pipet 10 ml of 100 mg/L stock solution into a 100 ml volumetric flask and fill it with water will produce the standard solution of 10 mg/L. Repeat the dilution step until the desired concentration range is obtained.

Record in the scientific notebook steps taken to make serial dilution (i.e., the volume pipetted and size of volumetric flasks).

3.2.3 Preparatory Steps of Liquid Samples

To analyze dissolved metals, filter the sample through a filter (e.g., 0.45-um) and acidify the liquid phase with concentrated HNO_3 (e.g., 5 ml/L). Record filtration and acidification steps in the scientific notebook.

3.2.4 Calibration and Sample Analysis Method

The calibration curve is obtained from the set of calibration standards (see Section 3.2.2). The detailed calibration and sample analysis method are described in Sections 7 and 8 of Attachments 1 and 2.

3.2.5 Identification of Calibration Intervals

Calibration shall be performed each day that samples are analyzed, according to Attachments 1 and 2.

3.2.6 Identification of Tolerances and Ranges of Use

Calibration curves shall be used to define the range of use and tolerances for M&TE covered by this TIP. The correlation coefficient (R^2) value (e.g., use the built-in functions in Microsoft Excel), of the calibration curve shall define the tolerance. In order for the calibration curve to be acceptable, the R^2 must be equal to or greater than 0.998.

3.2.7 M&TE Storage and Handling

M&TE shall not be handled in a manner that adversely affects its current or future performance. M&TE shall be used in laboratory environments, and stored at room temperature.

3.2.8 Calibration Documentation

In accordance with YAP-12.3Q, staff members shall document the following information in the scientific notebook or on the M&TE Calibration Documentation Form (Attachment 4) that includes, as a minimum, the information required:

a) The unique identification of the M&TE calibrated

b) Date calibrated

c) Calibration data

d) Recalibration due date or calibration interval / frequency

e) Procedure (including revision level) used to calibrate the M&TE

f) Identification of and traceability to the calibration standards used for the calibration

g) Results of the calibration and statement of acceptability

h) As-found condition of the M&TE

i) Specified range and tolerances and whether the M&TE met those tolerances

j) Personnel performing calibrations

k) Reference to actions taken with out-of-calibration or nonconforming M&TE, including evaluation results, as appropriate.

Calibration is required each day samples are analyzed, and calibration is an integral part of the measurement procedure. A calibration sticker containing the following information shall be affixed to the instrument.

Calibration
By: LBNL staff following the TIP for calibration.
This instrument shall be calibrated each day samples are analyzed.

Copies of the calibration results shall be provided to the LBNL M&TE Coordinator to update the M&TE list as per YAP-12.3Q.

3.2.9 Controls for Out-of-Calibration Conditions

If any out-of-calibration conditions (as described in YAP-12.3Q) are determined to exist for the M&TE (e.g., instrument produces results known to be in error), the instrument shall have an out-of-service tag applied indicating that it is not to be used and, when possible, the instrument shall be moved to a segregated “out-of-service” area.

The above conditions shall be documented by using the M&TE Out of Calibration Report (OCR) in accordance with the instructions provided in YAP-12.3Q. If it is determined that the data is impacted, a Nonconformance Report (NCR) shall be initiated in accordance with YAP-15.1Q, *Control of Nonconformances*.

3.2.10 Recalibration When Updates to Software Contained Affects Calibration

A routine might be used for automatic correction of spectral interference (e.g., Section 3.1.2 of Method 6010B). All software, including this routine, is supplied from the vendor as an integral part of the instrument and as such is controlled by calibrating the M&TE in accordance with YAP-12.3Q. Software updates will not affect the previous calibrations as calibration is required each day when samples are analyzed.

3.2.11 Staff Member shall document each usage of the equipment in the M&TE Standard Usage Log or scientific notebook containing the same information as the M&TE Standard Usage Log, as described in YAP-12.3Q.

4. RECORDS

4.1 Lifetime

Records generated as a result of this TIP are entries in:

- Scientific notebooks or attachments to such notebooks,
- Equipment Logbooks (including M&TE Justification Forms and Standard Usage Log, if applicable) controlled as supplemental records to the scientific notebook,
- M&TE Out of Calibration Reports, if applicable.

4.2 Non-Permanent

None

4.3 Controlled Documents

This Technical Implementing Procedure

4.4 Records Center Documents

Records associated with this procedure shall be submitted to the Records Coordinator for transmittal to the Records Processing Center (RPC) in accordance with AP-17.1Q, *Record Source Responsibility for Inclusionary Records*.

5. RESPONSIBILITIES

5.1 The Principal Investigator (PI) is responsible for assuring full compliance with this procedure and providing training thereof. The PI is responsible for overseeing and coordinating the preparation, review, distribution, revision, and recommending rescission of the TIP.

5.2 Staff Members are responsible for following this procedure and turning over related documentation to the Records Coordinator for submittal to the RPC in accordance with AP-17.1Q. Related data shall be turned over to Technical Data Coordinator for entry into the YMP Technical Database Management System (TDMS) in accordance with YMP-LBNL-QIP-SIII.3, *Submitting Key Data to the Yucca Mountain Project Office*.

6. ACRONYMS AND DEFINITIONS

6.1 Acronyms

AP	OCRWM Administrative Procedure
EA	Engineering Assurance
EML	Environmental Measurements Laboratory at LBNL
EPA	Environmental Protection Agency
ICP-AES	Inductively Coupled Plasma– Atomic Emission Spectrometry
ICP-MS	Inductively Coupled Plasma–Mass Spectrometry
LBNL	Lawrence Berkeley National Laboratory
M&TE	Measuring and Test Equipment
MSDS	Material Safety Data Sheet
NCR	Nonconformance Report
NIST	National Institute of Standards and Technology
OCR	Out of Calibration Report
OCRWM	Office of Civilian Radioactive Waste Management
OQA	Office of Quality Assurance
PI	Principal Investigator
QARD	Quality Assurance Requirements and Description

QIP	Quality Implementing Procedure
QSL	Qualified Suppliers List
RPC	Records Processing Center
SMF	Sample Management Facility
TDMS	Technical Data Management System
TIP	Technical Implementing Procedure
YAP	YMP Administrative Procedure
YMP	Yucca Mountain Site Characterization Project

6.2 Definitions

Staff Member: Any scientist, engineer, research or technical associate, technician, or student research assistant performing quality-affecting work for YMP-LBNL.

Technical Implementing Procedure: Each TIP describes YMP-LBNL technical and/or scientific tasks that (1) are repetitive, (2) are standardized, and (3) can return different results if deviation from the sequence of steps occur.

7. REFERENCES

AP-2.16Q, *Activity Evaluation*

AP-17.1Q, *Record Source Responsibility for Inclusionary Records*

AP-SIII.1Q, *Scientific Notebooks*

AP-SIII.3Q, *Submittal and Incorporation of Data to the Technical Data Management System*

YAP-12.3Q, *Control of Measuring and Test Equipment and Calibration Standards*

YAP-15.1Q, *Control of Nonconformances*

YAP-SII.1Q, *Submittal, Review, and Approval of Requests for Yucca Mountain Site Characterization Project Geologic Specimens*

YMP-LBNL-QIP-5.2, *Preparing Development Plans & Quality/Technical Implementing Procedures*

YMP-LBNL-QIP-SII.0, *Documenting Sample Control*

8. ATTACHMENTS

Attachment 1: Method 6010B, Inductively Coupled Plasma-Atomic Emission Spectroscopy, EPA SW-846. Sentences and/or sections that are crossed out are irrelevant and/or addressed specifically in the TIP.

Attachment 2: Method 6020, Inductively Coupled Plasma-Mass Spectrometry, EPA SW-846. Sentences and/or sections that are crossed out are irrelevant and/or addressed specifically in the TIP.

Attachment 3: Chain of Custody Record.

Attachment 4: M&TE Calibration Documentation Form.

9. REVISION HISTORY

06/30/00, Revision 0, Modification 0

Initial issue.

10. APPROVAL

Signature on file

Preparer: Qinhong (Max) Hu Date:

Signature on file

Technical Review: H. Scott Mountford Date:

Signature on file

Technical Review: Timothy J. Kneafsey Date:

Signature on file

EA Reviewer: Nancy Aden-Gleason Date:

Signature on file

OQA Concurrence: Stephen Harris Date:

Signature on file

Principal Investigator: Joseph S.Y. Wang Date:

Signature on file

Project Manager: Gudmundur S. Bodvarsson Date:

YMP-LBNL-TIP/AFT 11.0, Rev. 0, Mod. 0
Attachment 1

Method 6010B, Inductively Coupled Plasma-Atomic Emission Spectrometry, EPA SW-846

This document is not available on the YMP-LBNL website. A copy can be downloaded from the EPA website:

http://www.epa.gov/epaoswer/hazwaste/test/main.htm

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YMP Activity:	**Chain of Custody Record**	Chain-of-Custody Number:

Send Results to: MS:

Telephone: Fax:

Sampler Name(s):

Sample Number	Date	Time	Sample Matrix	Preservative	Type and Number of Containers	ANALYSES	Date Results Required	Remarks, Instructions and/or Special Requests

Relinquished by: (signature)	Date/Time	Received by: (signature)	Relinquished by: (signature)	Date/Time	Received by: (signature)
Relinquished by: (signature)	Date/Time	Received by: (signature)	Relinquished by: (signature)	Date/Time	Received by: (signature)
Relinquished by: (signature)	Date/Time	Received at Environmental Measurement Laboratory by: (signature)		Date/Time:	

M&TE Calibration Documentation Form

a) M&TE description	**b) M&TE unique identification**	**c) Calibration date and time (if applicable)**
d) Person performing calibrations		**e) M&TE condition (as-found)** Working _____ Not working _____
f) Calibration procedure (including revision level)		**g) Calibration standards used**
h) Location of calibration data YMP-LBNL-_____ Page(s):		**i) Location of calibration results** YMP-LBNL-_____ Page(s):
j) Specified range and tolerances		
k) Statement of acceptability including acceptability of range and tolerances Range acceptable Yes _____, No _____ Tolerance acceptable Yes _____, No _____ Calibration acceptable Yes _____, No _____ Comments:		
l) Re-calibration due date or calibration interval/frequency		**m) Reference to actions taken with out-of-calibration or non conforming M&TE, including evaluation results, as appropriate** YMP-LBNL-_____ Page(s):
n) Comments		

Signature Date